



New Results from the Scanning Electron Microscopy Characterization of Fuel Plates Irradiated in the EMPIrE Irradiation Experiment

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Reduced Enrichment for
Research and Test Reactors
Conference

Vienna, Austria

October 3-5, 2022

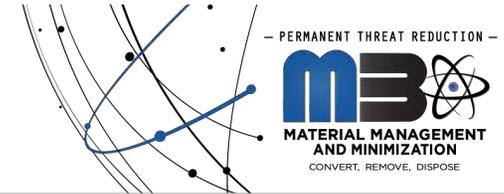
— PERMANENT THREAT REDUCTION —



**MATERIAL MANAGEMENT
AND MINIMIZATION**

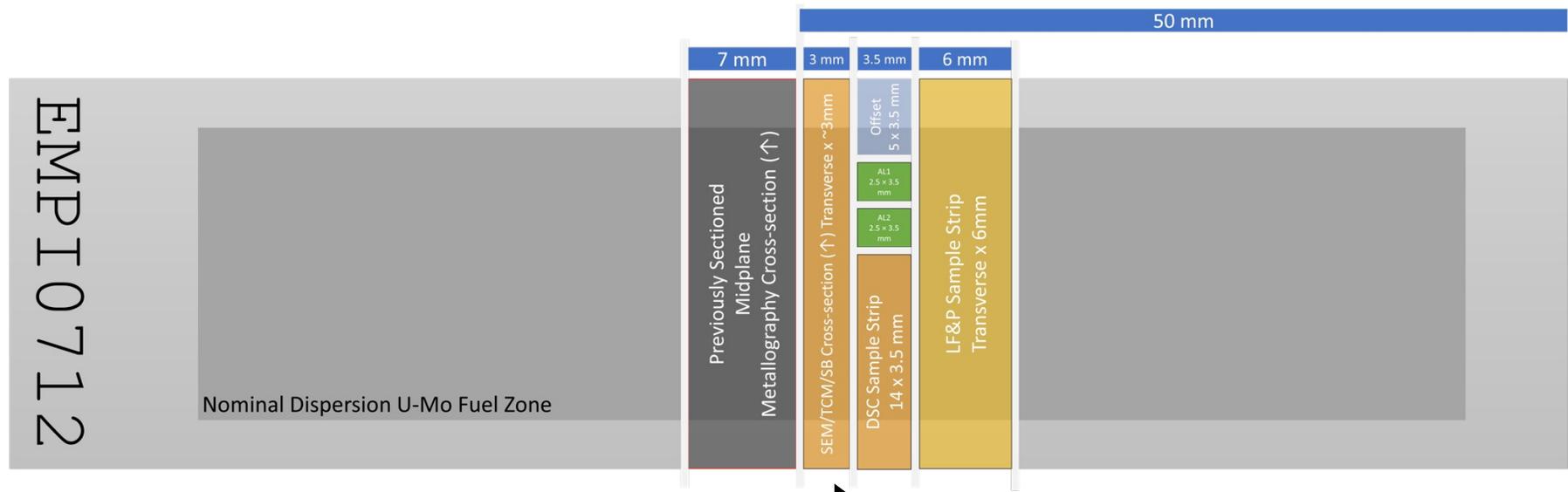
CONVERT, REMOVE, DISPOSE

Introduction



- Irradiation of the EMPIRE experiment has been completed in the Advanced Test Reactor, and microstructural characterization of the irradiated fuel plates is underway. This talk focuses on new scanning electron microscopy (SEM) analysis results for fuel plates that have been characterized.
- In the Hot Fuel Examination Facility (HFEF), cross sections were taken from plate midplane for fuel plates EMPI 0702, 0712, and 0905.
 - Two fuel plates (0702 and 0712) had around a 2 micron-thick PVD ZrN coating, as-atomized U-7Mo powder with no heat treatment, and zero fines.
 - One fuel plate (0905) had around a 2 micron-thick PVD ZrN coating, heat treated U-7Mo powder, modified powder distribution, and used CERCA powder
 - Plates were irradiated in ATR in the south flux trap face-on to the flux, so the neutron flux gradient was relatively flat across each cross section.
 - Optical metallography (OM) performed on cross sections.
 - Separate cross sections produced for SEM analysis.
- Sample cross-sections transferred to the Irradiated Materials Characterization Laboratory (IMCL), where they were mounted and polished.
- SEM was performed to generate backscattered electron (BSE) images at magnifications up to 5,000x.
 - A focused ion beam (FIB) was employed to produce three samples, about 50 microns long, from each fuel plate for composition analysis using energy dispersive spectroscopy (EDS) and wavelength dispersive spectroscopy (WDS).
 - FIB samples captured regions of the U-7Mo fuel particle and the fuel particle/coating and coating/Al matrix interfaces.
- Microstructural characterization results for Type 7A and Type 8A fuel plates are being compared to better understand how some of the different variables being tested in the EMPIRE experiment affect fuel performance.

Nominal Sectioning Plan



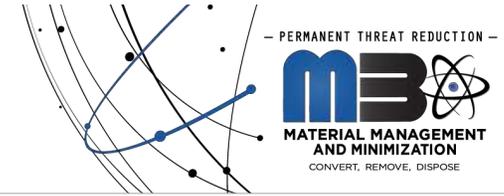
EMPI 0712

Nominal Dispersion U-Mo Fuel Zone

50 mm

Cross-section for SEM

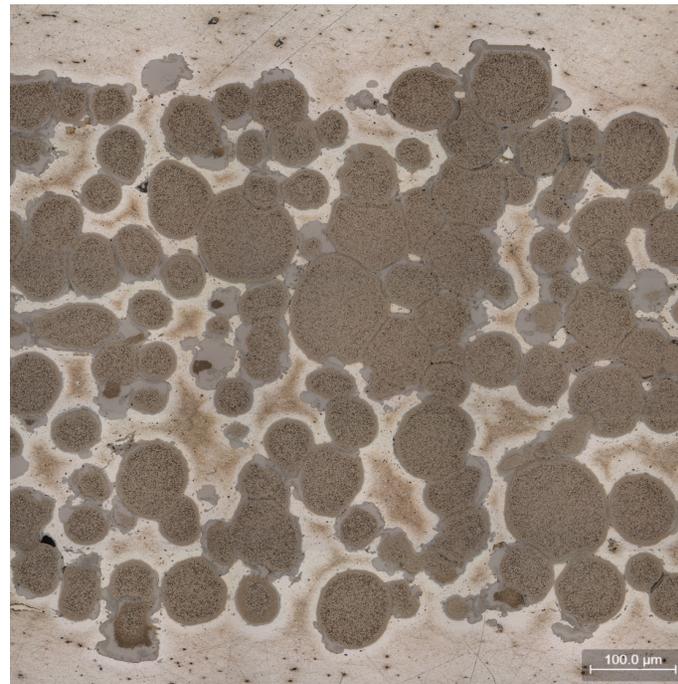
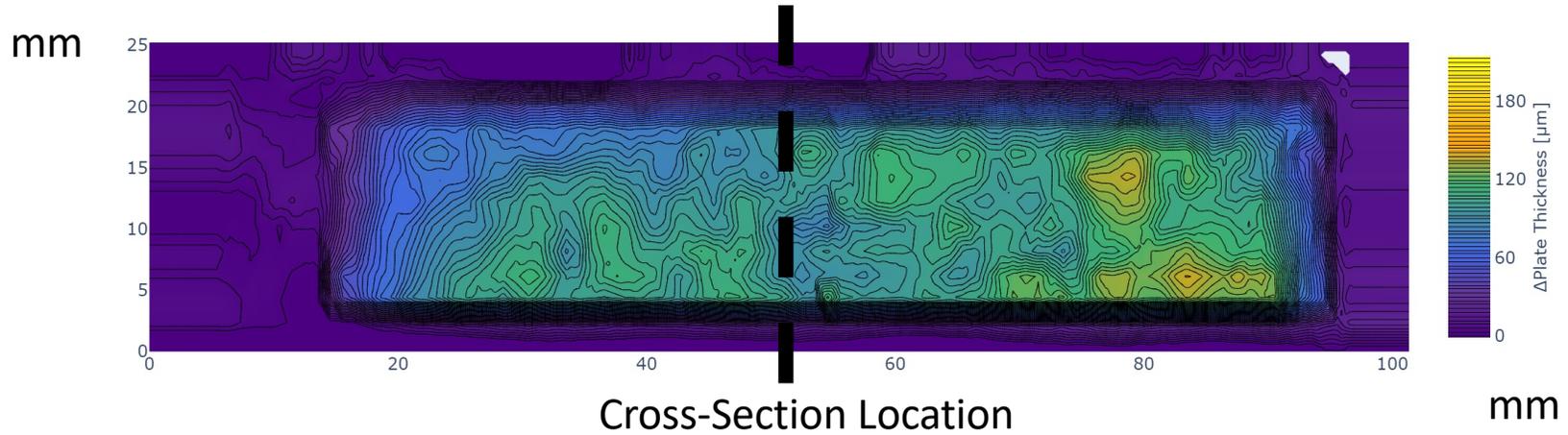
Calculated EMPI 0702, 0712, 0905 Irradiation Details



Sample	SEM Cross-Section Fission Density (FD) Minimum (fissions/cm ³ UMo)	SEM Cross-Section FD Average (fissions/cm ³ UMo)	SEM Cross-Section FD Maximum (fissions/cm ³ UMo)	Fuel Plate BOL Average Surface Heat Flux (W/cm ²)	Fuel Plate BOL Peak Surface Heat Flux (W/cm ²)
EMPI 0702	5.6 x10 ²¹	5.7 x10 ²¹	5.8 x10 ²¹	448.9	513.4
EMPI 0712	2.9 x10 ²¹	3.0 x10 ²¹	3.2 x10 ²¹	446.8	520.9
EMPI 0905	6.0 x10 ²¹	6.1 x10 ²¹	6.2 x10 ²¹	474.0	544.1

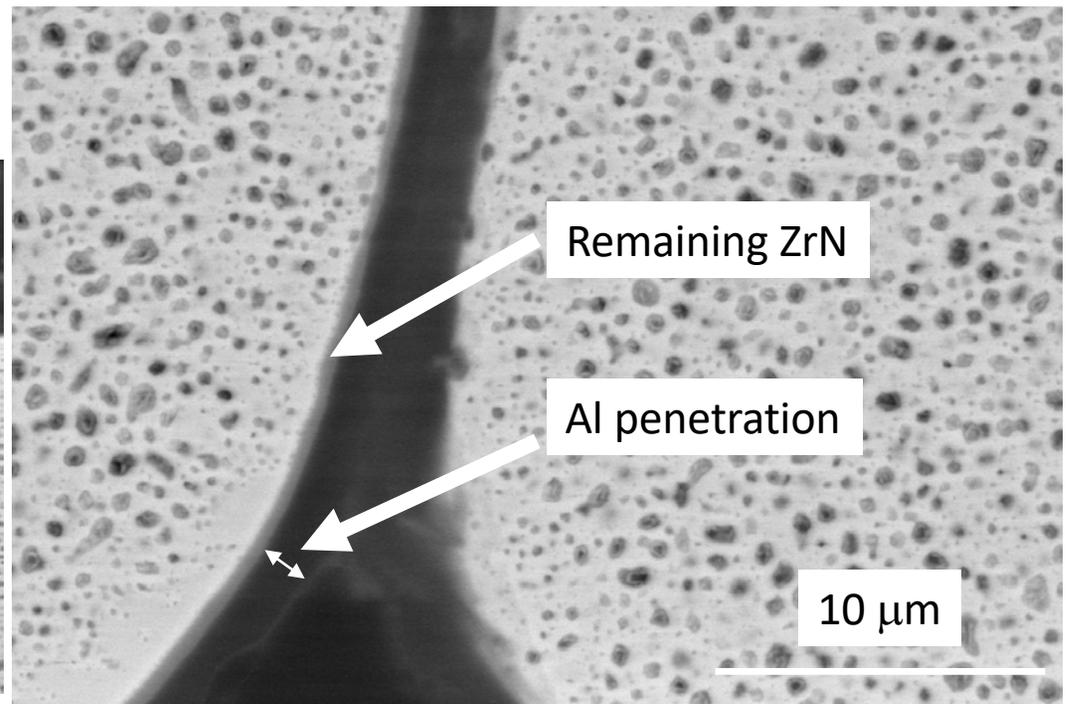
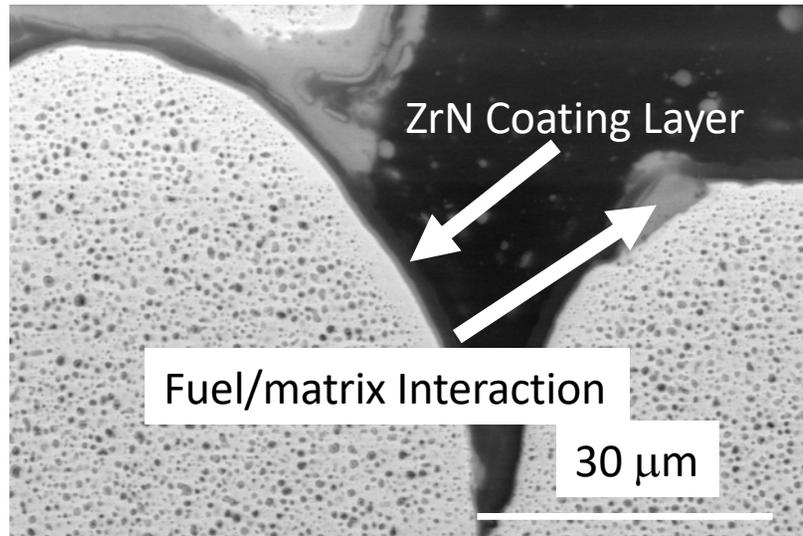
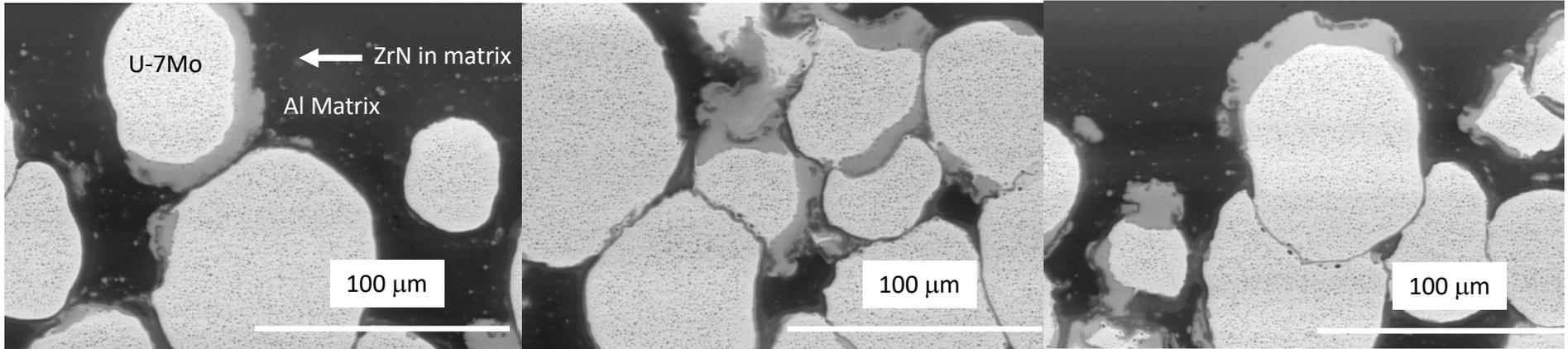
Note: Fission density values for analyzed SEM cross-section, and surface heat flux values are for fuel plate.

EMPI0702 - ZrN PVD coating with no U-7Mo powder heat treatment (OM) 285 W/cm² Peak Heat Flux, 79% Burnup

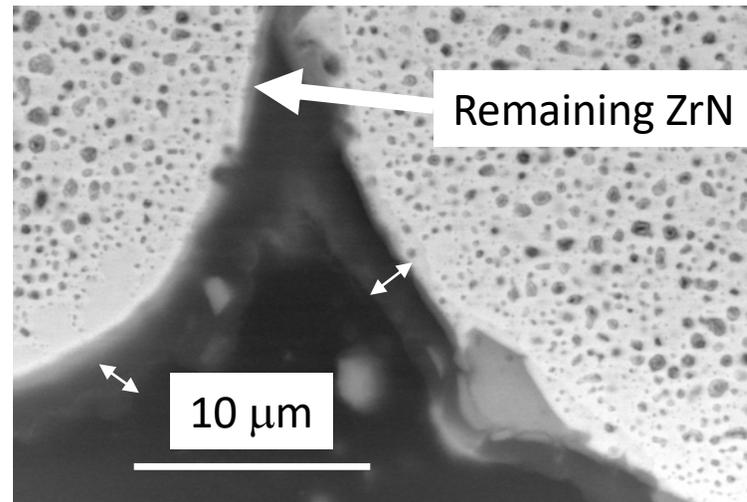
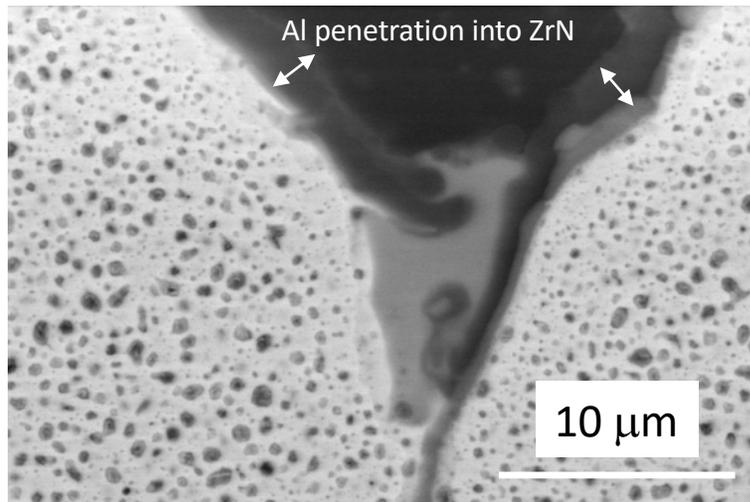
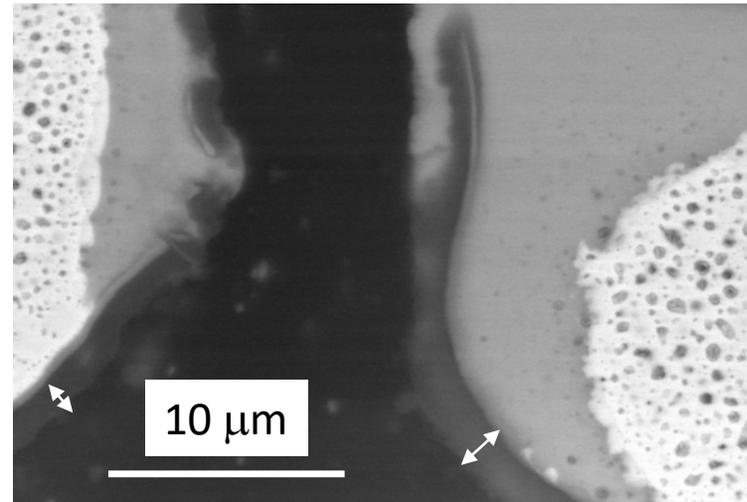
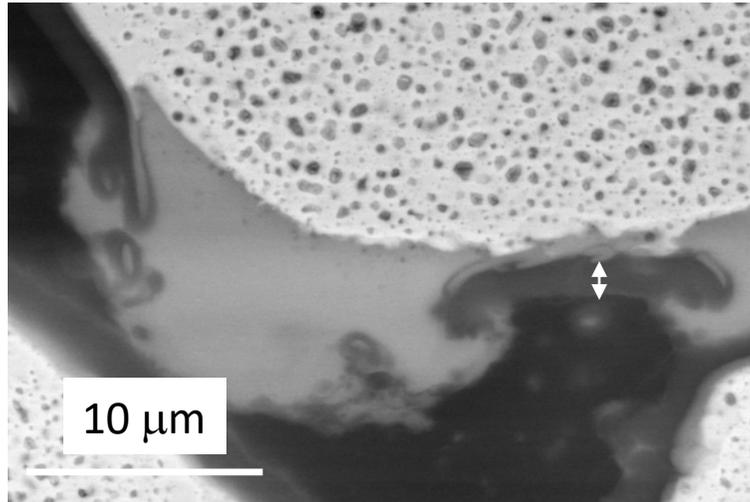


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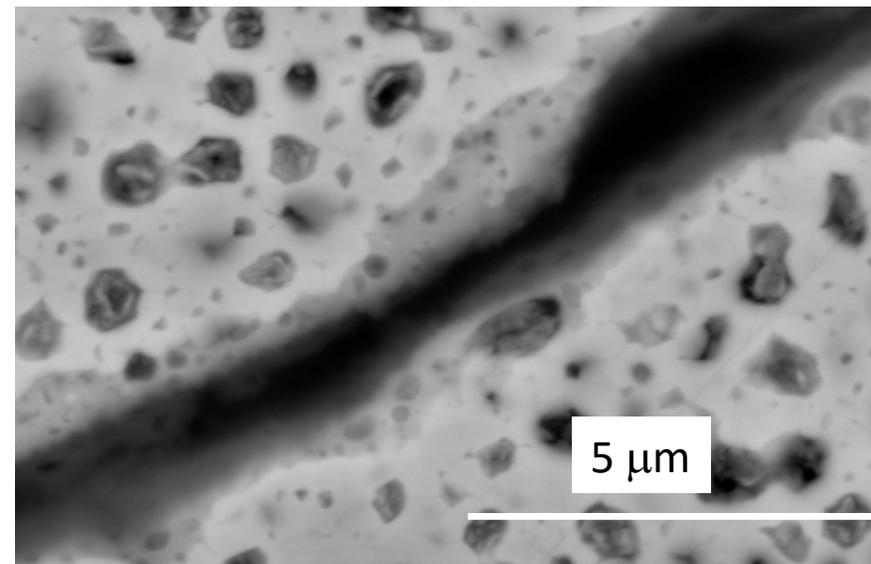
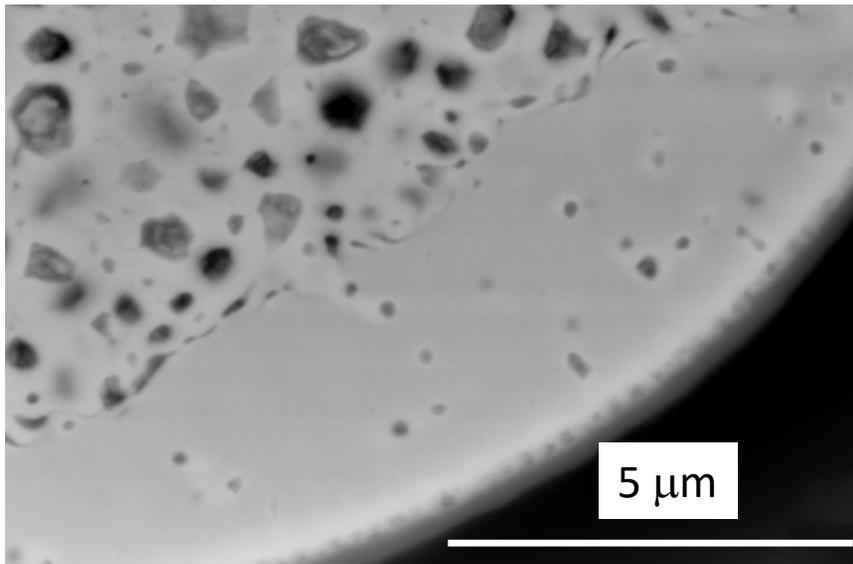
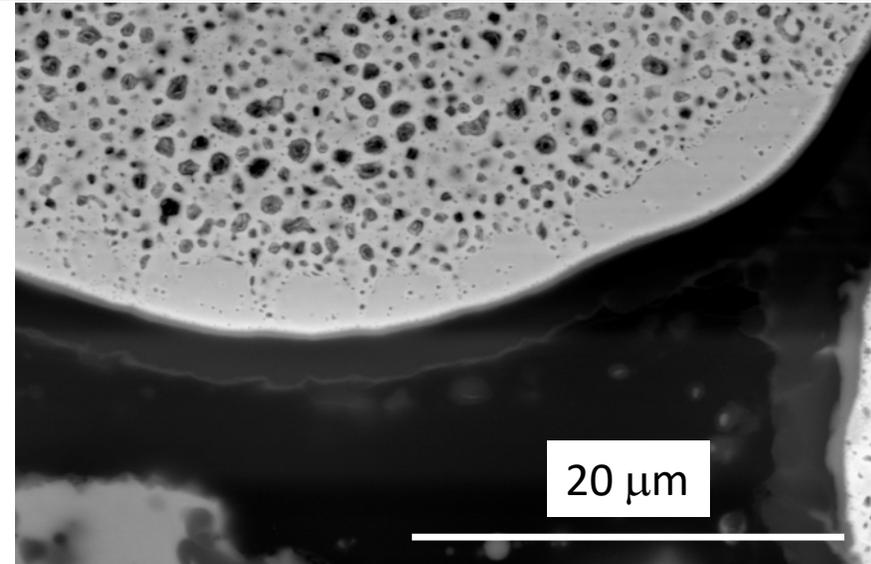
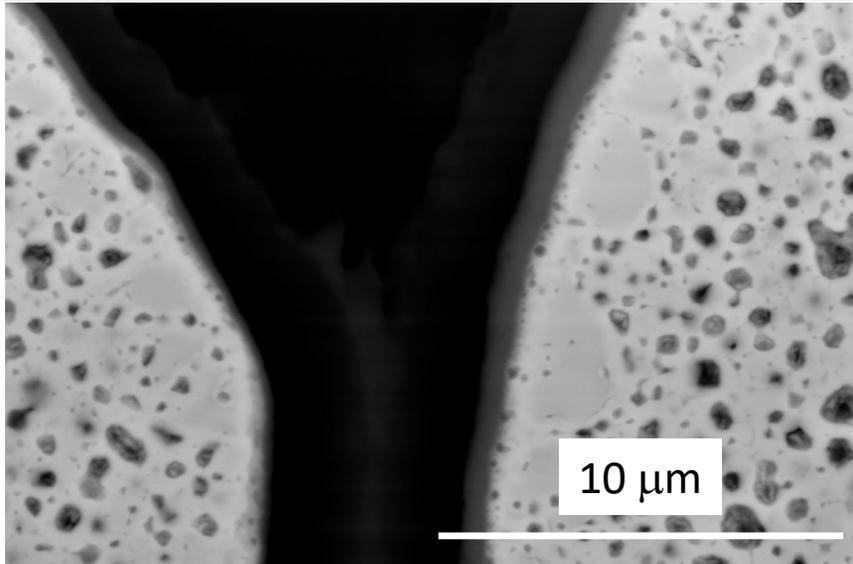
*EMPI0702 - ZrN PVD coating with no U-7Mo powder heat treatment (SEM)
513 W/cm² Peak Heat Flux, 5.7x10²¹ fissions/cm³ Average Fission Density*



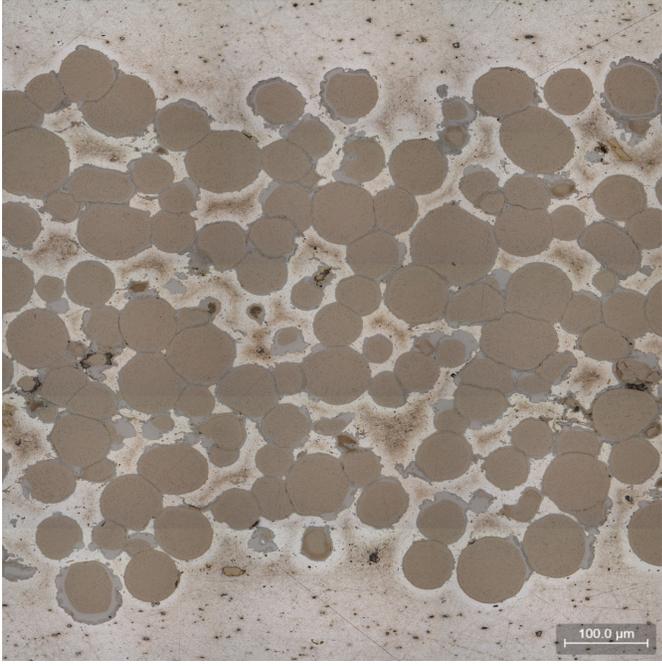
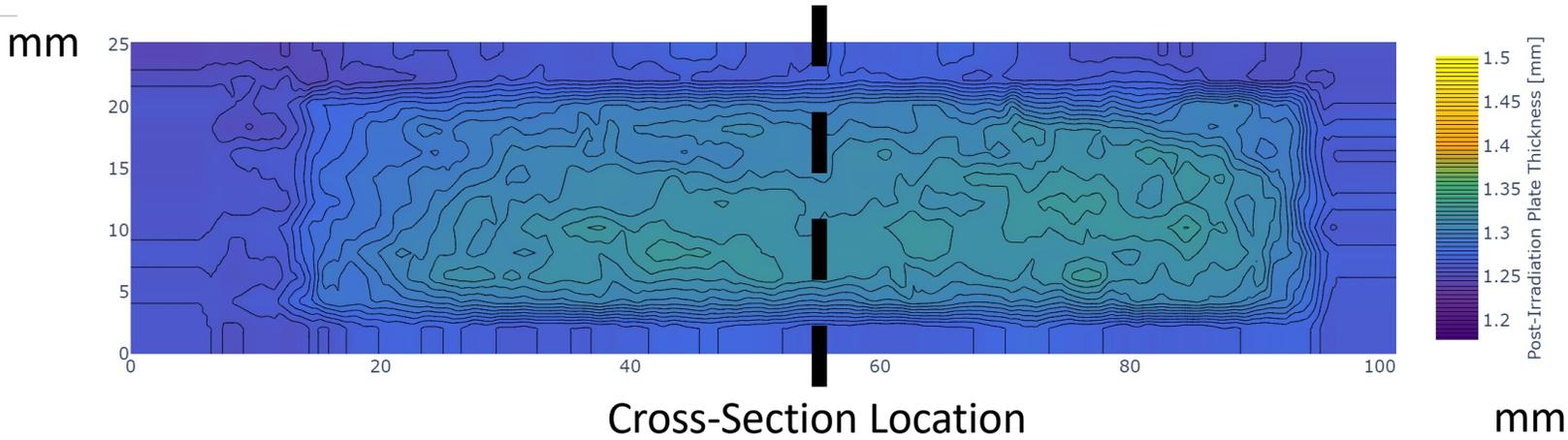
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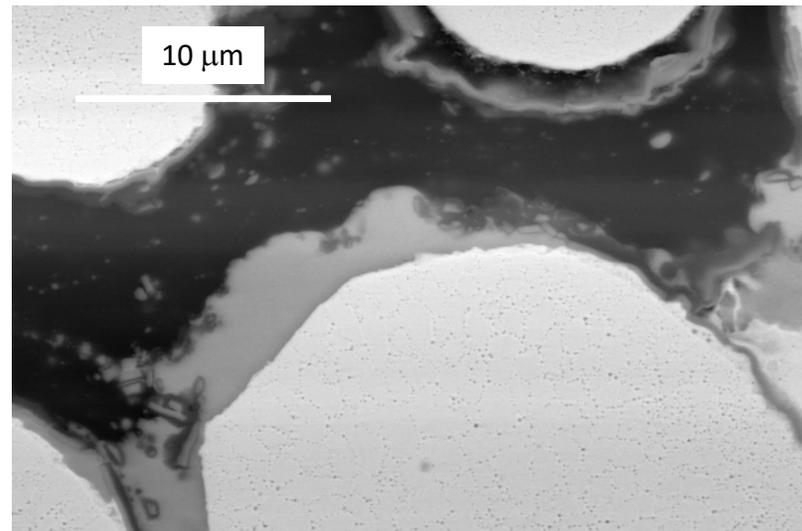
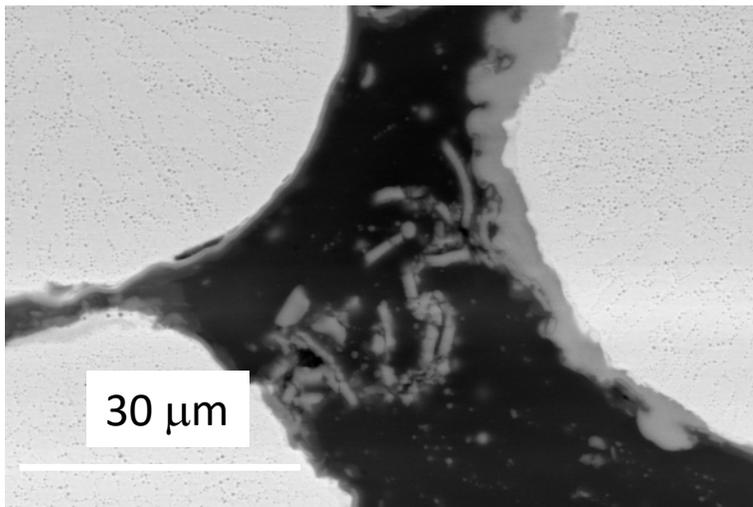
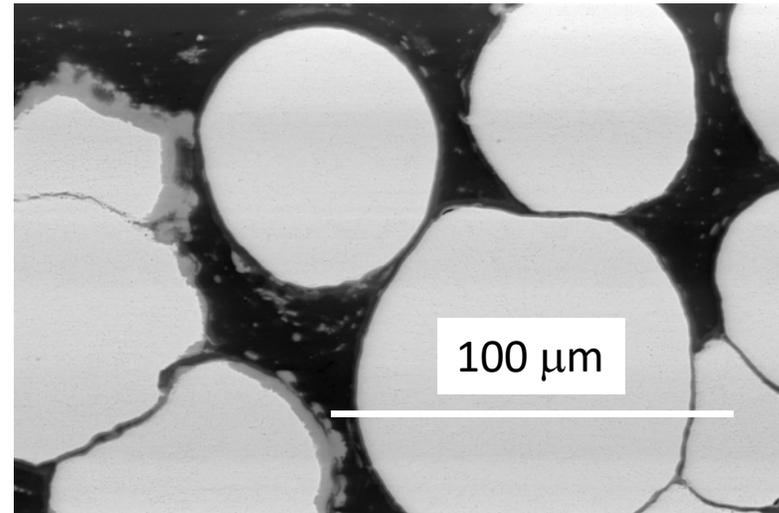
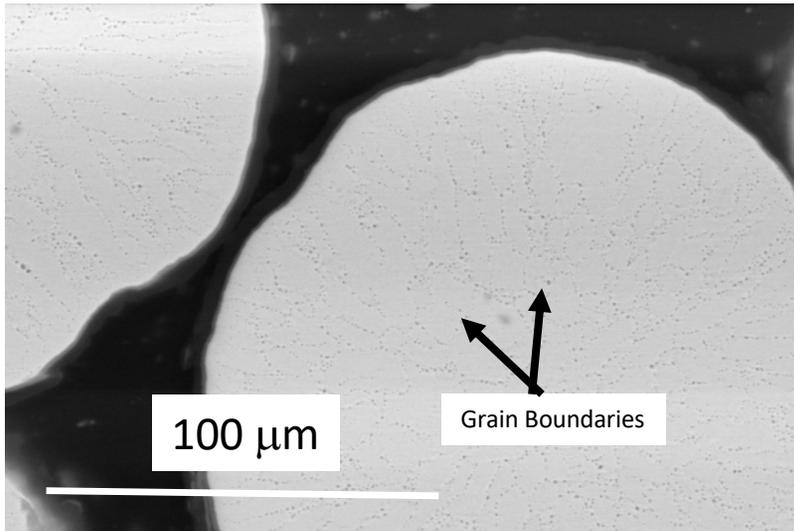


*EMPI0712 - ZrN PVD coating with no U-7Mo powder heat treatment (OM)
521 W/cm² Peak Heat Flux, 3.0x10²¹ fissions/cm³ Average Fission Density*

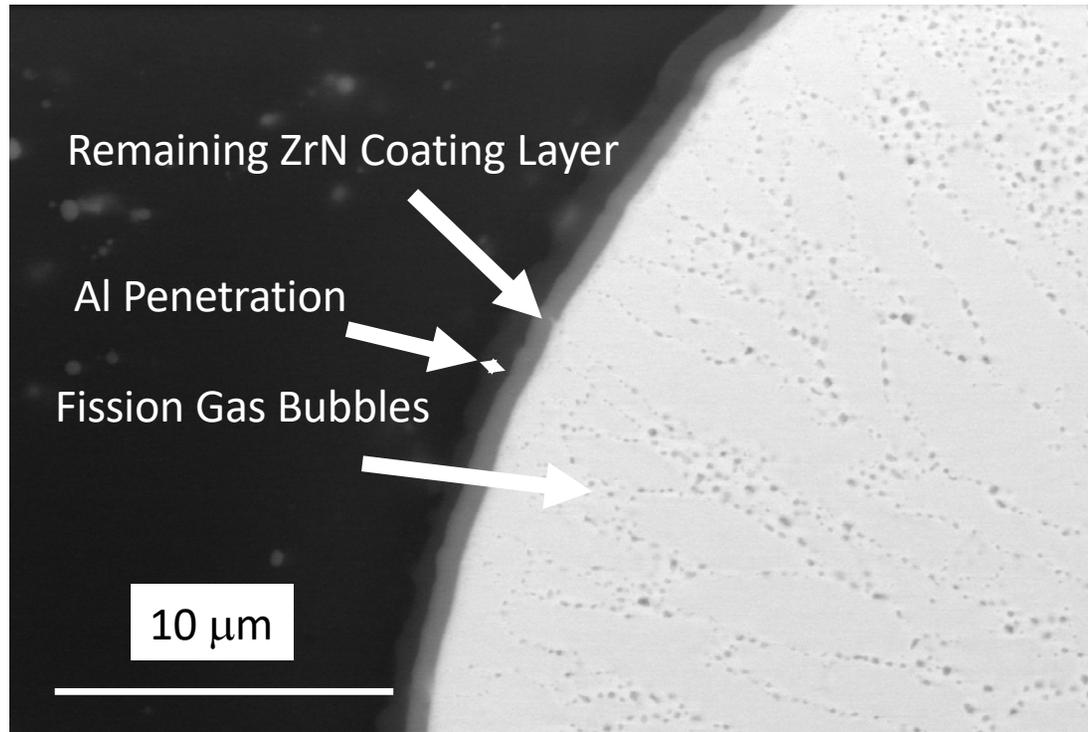


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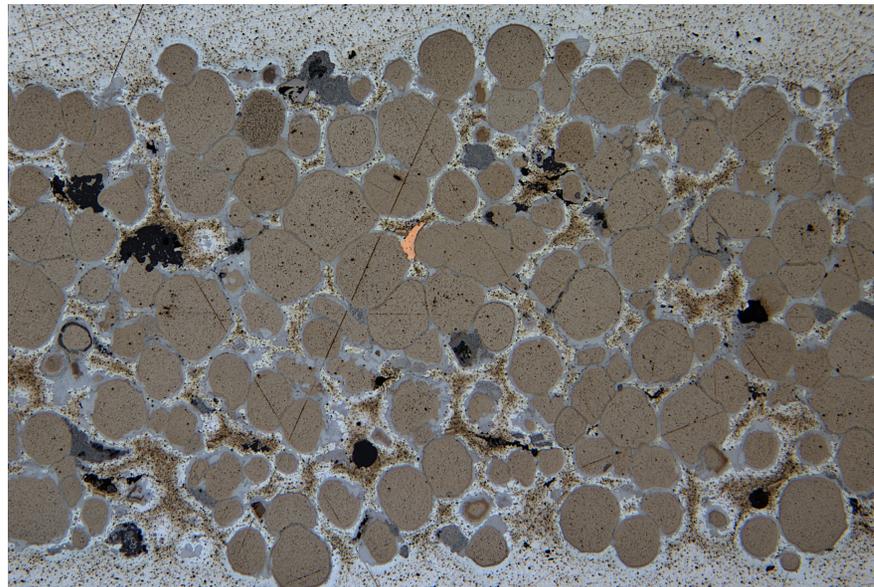
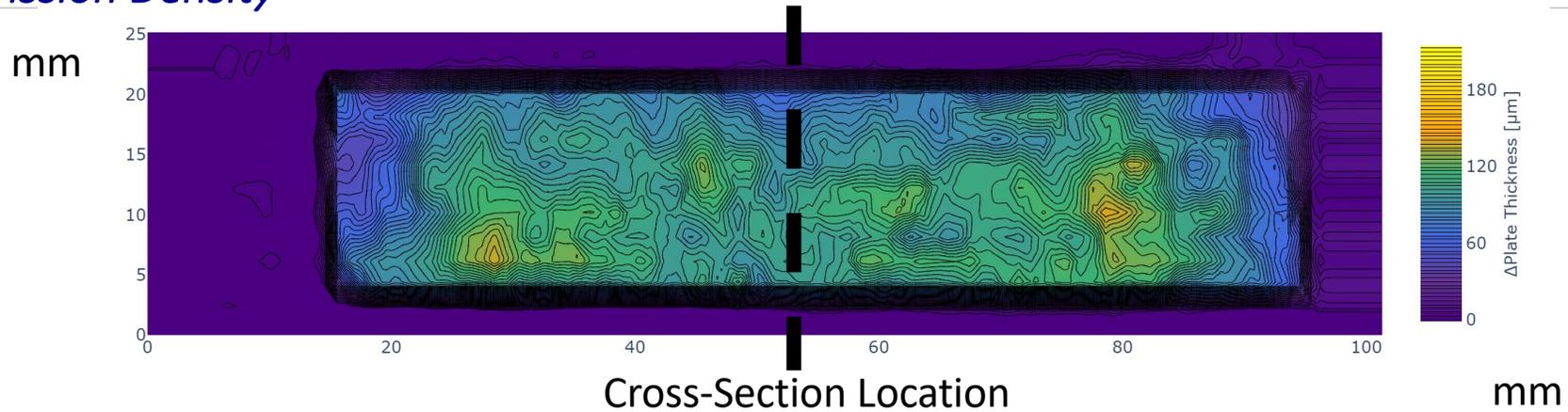
*EMPI0712 - ZrN PVD coating with no U-7Mo powder heat treatment (OM)
521 W/cm² Peak Heat Flux, 3.0x10²¹ fissions/cm³ Average Fission Density*



*EMPI0712 - ZrN PVD coating with no U-7Mo powder heat treatment (OM)
521 W/cm² Peak Heat Flux, 3.0x10²¹ fissions/cm³ Average Fission Density*

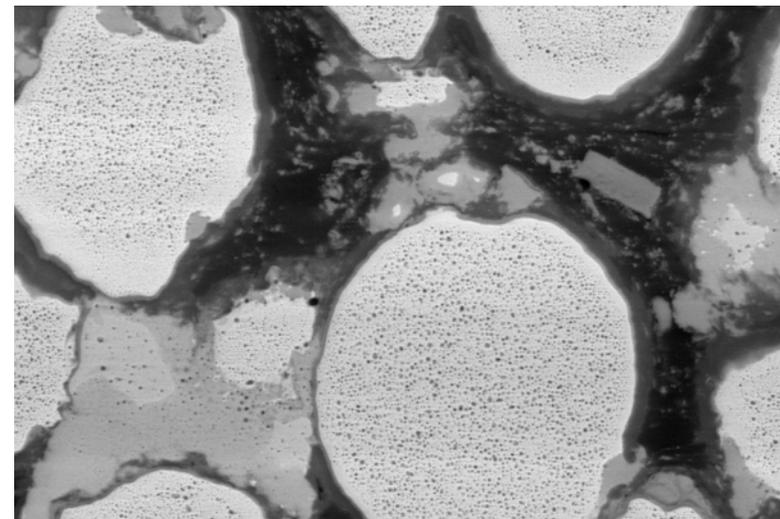
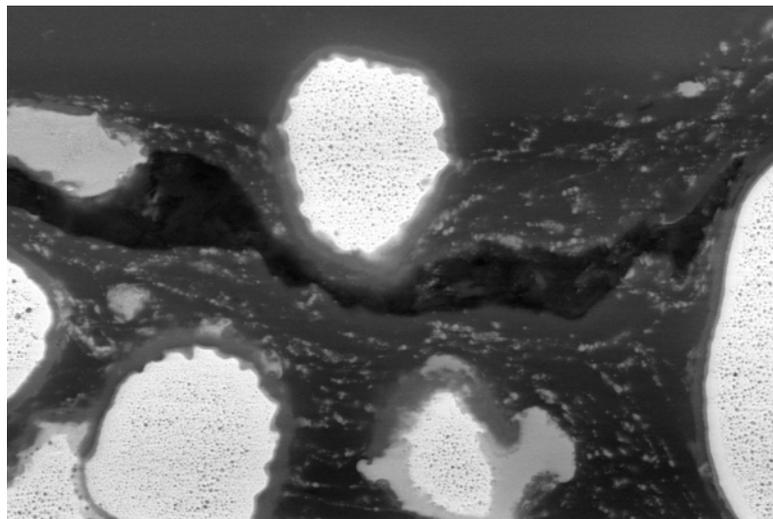
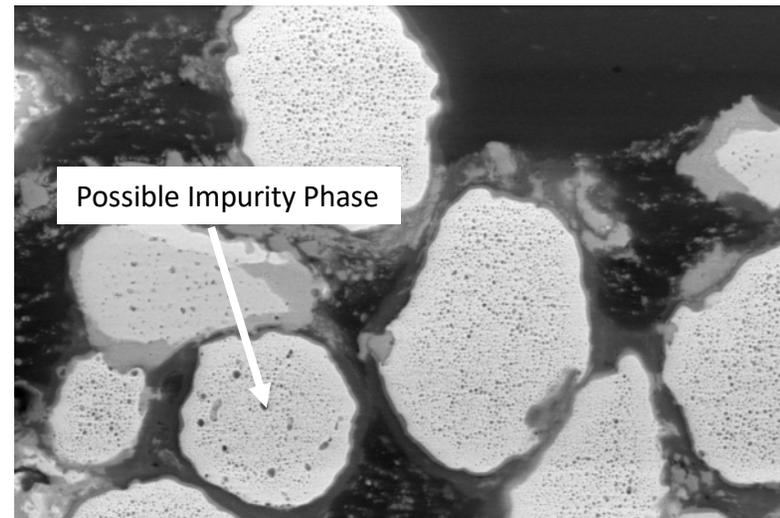
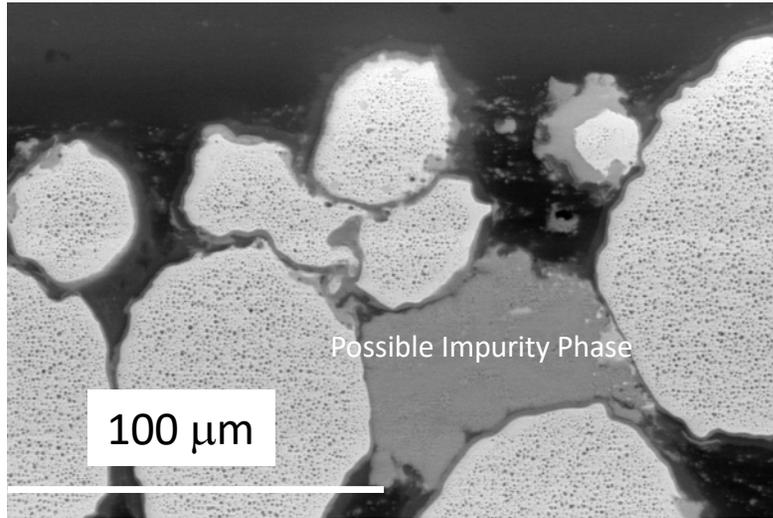


*EMPI0905 - ZrN PVD coating with U-7Mo powder heat treatment (OM),
CERCA Powder, 544 W/cm² Peak Heat Flux, 6.1x10²¹ fissions/cm³ Average
Fission Density*

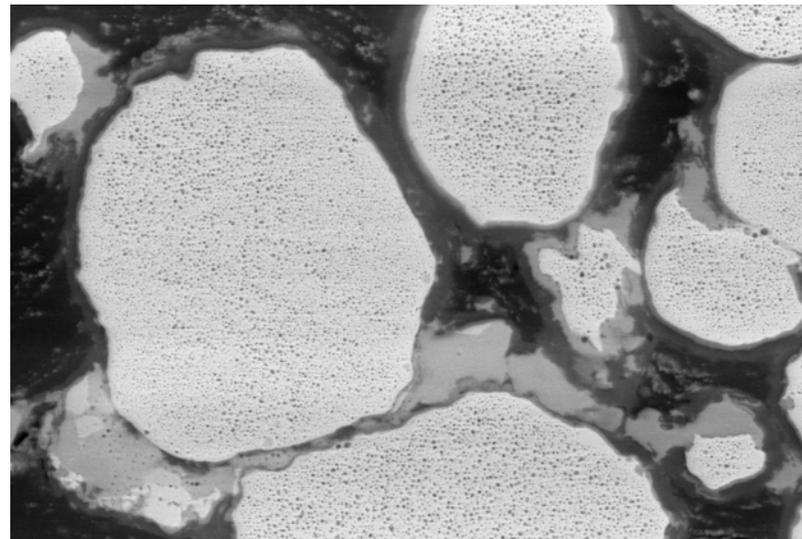
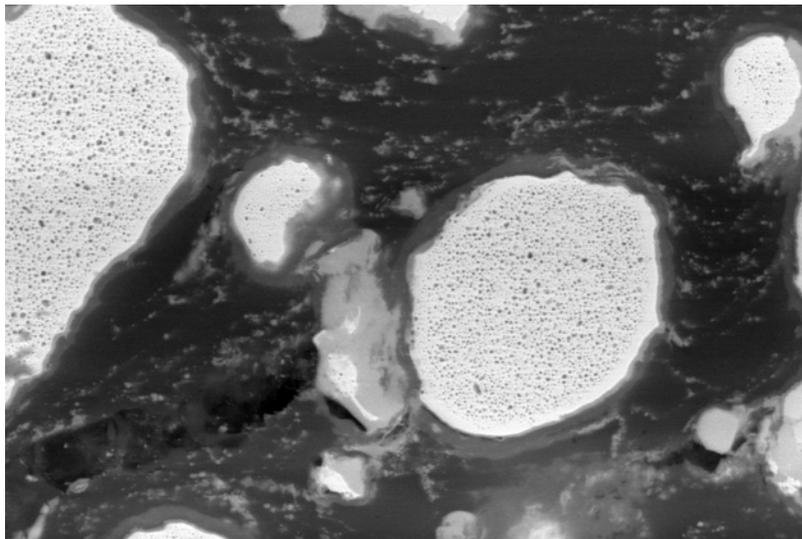
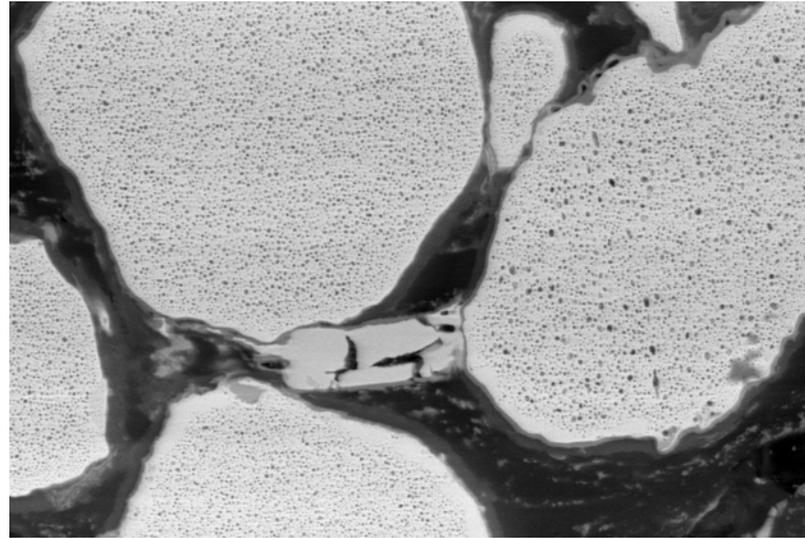
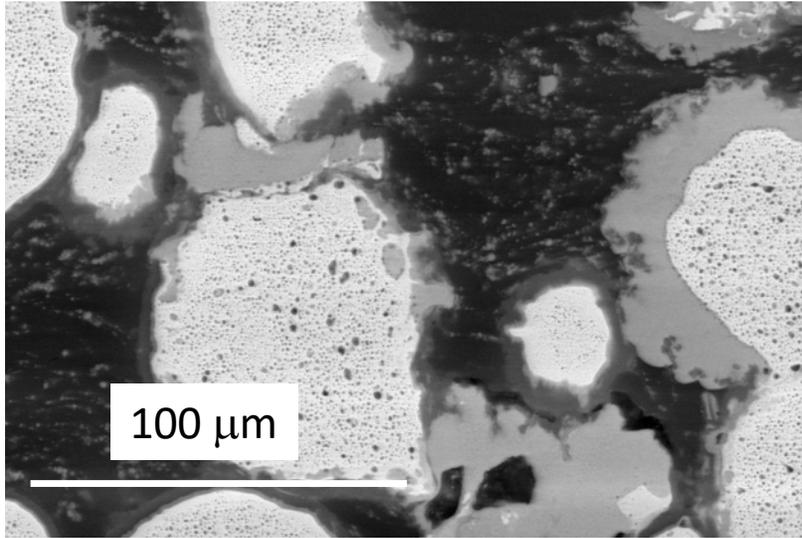


50x

EMPI0905 - ZrN PVD coating with U-7Mo powder heat treatment (OM), CERCA Powder, 544 W/cm² Peak Heat Flux, 6.1x10²¹ fissions/cm³ Average Fission Density (1kx magnification)

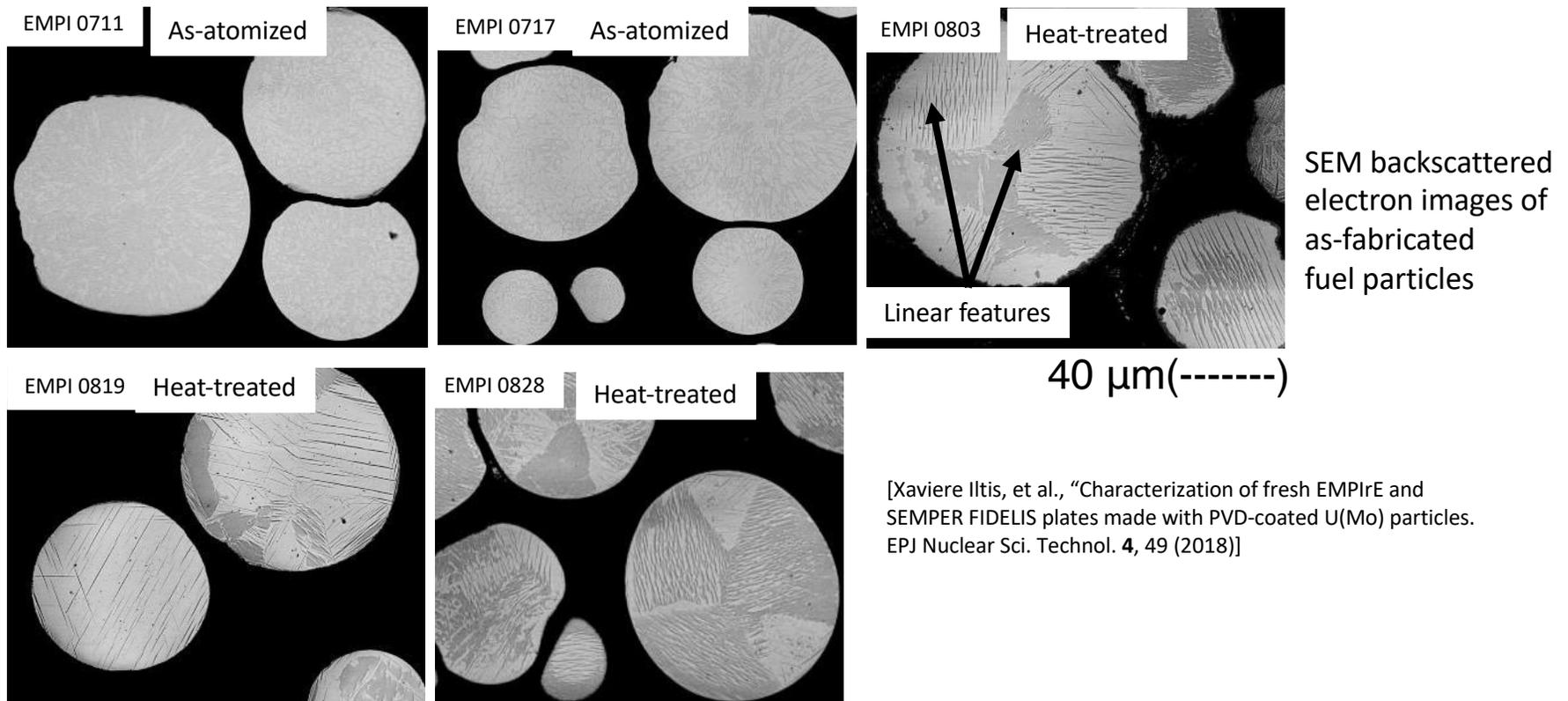


EMPI0905 - ZrN PVD coating with U-7Mo powder heat treatment (OM), CERCA Powder, 544 W/cm² Peak Heat Flux, 6.1x10²¹ fissions/cm³ Average Fission Density (1kx magnification)

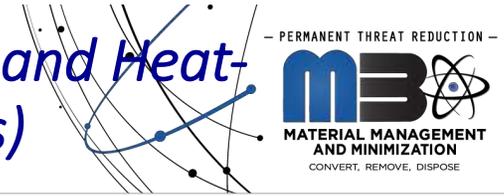


Comparison of Irradiation Behavior of As-Atomized and Heat-Treated U-7Mo Powder: (Starting Microstructure)

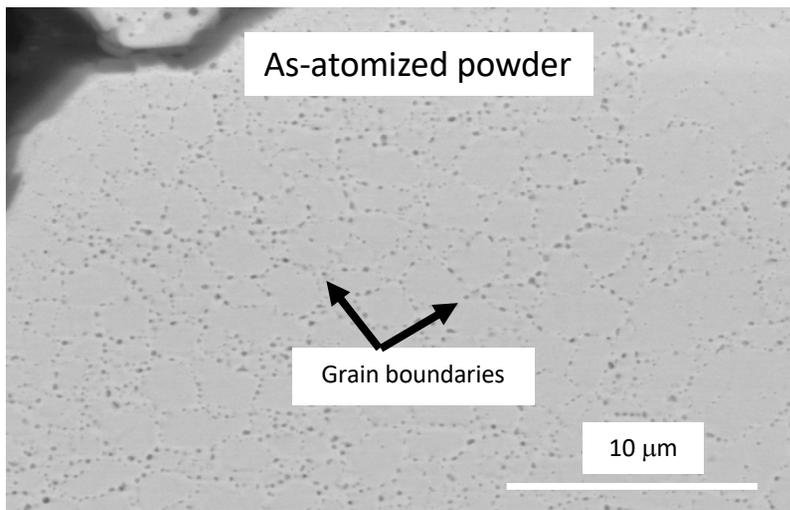
- Comparisons can be made of U-7Mo microstructures for Type 7A fuel plates to those in Type 8A fuel plates.
 - Powders heat-treated for 1,000°C for 1 hour employed in fuel plates EMPI 0818, 0820, and 0821.
 - As-atomized powders employed in fuel plates EMPI 0702 and 0712 fuel plates.
- The starting heat-treated powders exhibited large grains (tens of microns in size) with lamellar features that were interpreted as Widmanstatten microstructures that form due to rapid cooling.
 - As-atomized particles exhibit columnar microstructures that had a grain size of a few microns.
- The Mo content for the fuel particles was measured using SEM/EDS to be around 6.3 wt.%.



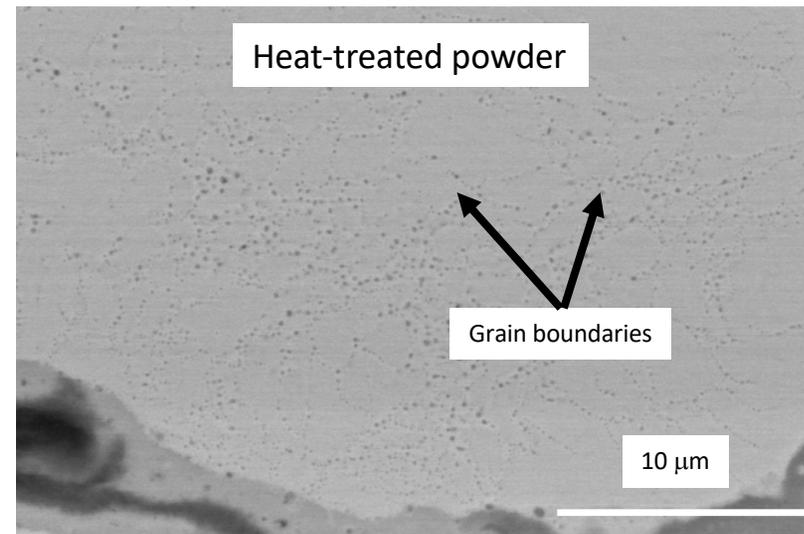
Comparison of Irradiation Behavior of As-Atomized and Heat-Treated U-7Mo Powder: (Irradiated Microstructures)



- Fuel plates EMPI 0712 and EMPI 0818 irradiated under similar conditions and exhibit similar U-7Mo microstructures

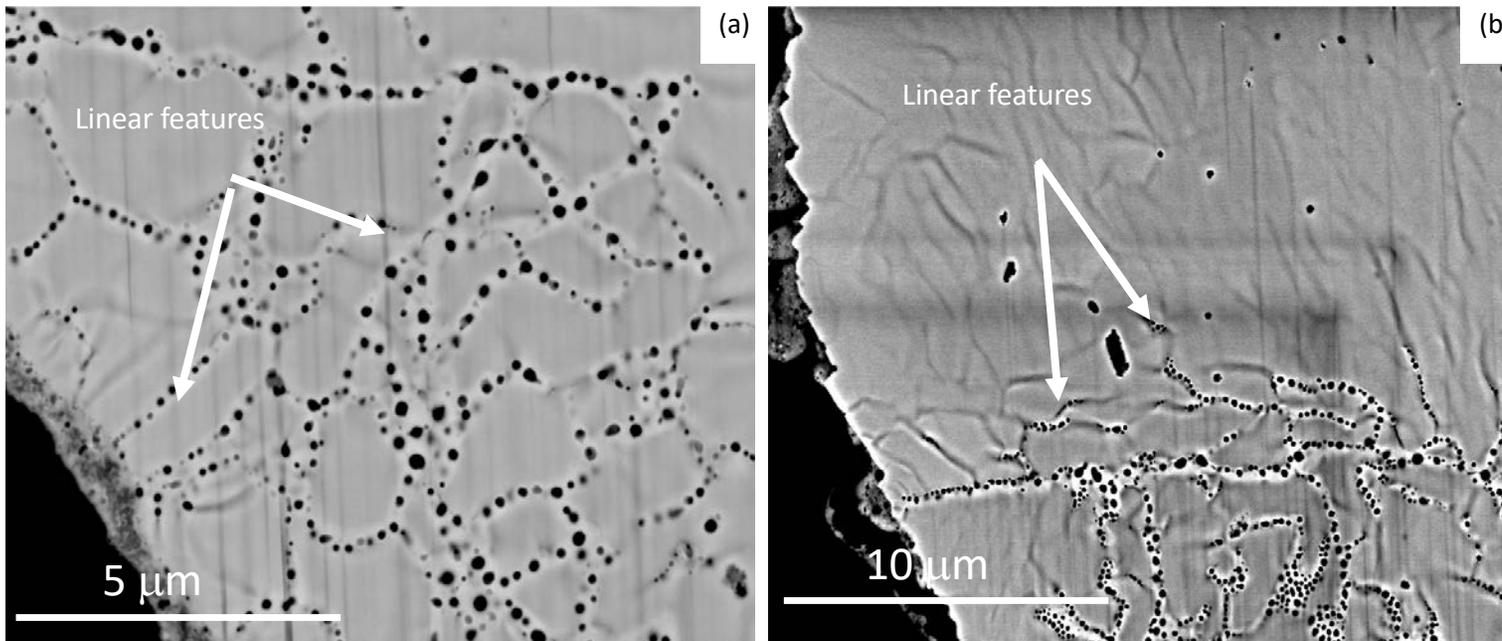


EMPI 0712 (512 W/cm² peak heat flux, 3.0x10²¹ fissions/cm³ average fission density)



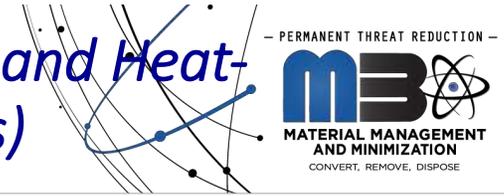
EMPI 0818 (522 W/cm² peak heat flux, 3.3x10²¹ fissions/cm³ average fission density)

Comparison of Irradiation Behavior of As-Atomized and Heat-Treated U-7Mo Powder: (Irradiated Microstructures)

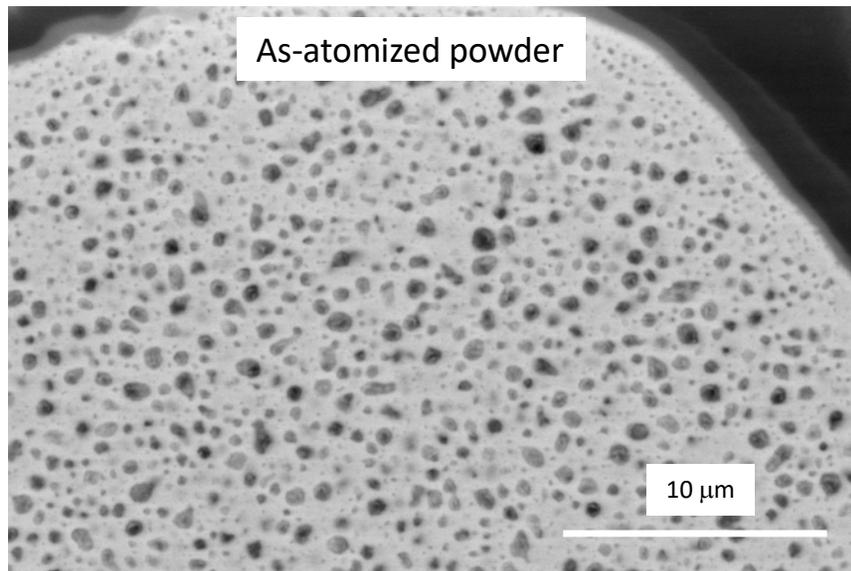


BSE images of samples produced using a focused ion beam from heat treated U-7Mo fuel particles irradiated in fuel plate EMPI 0818

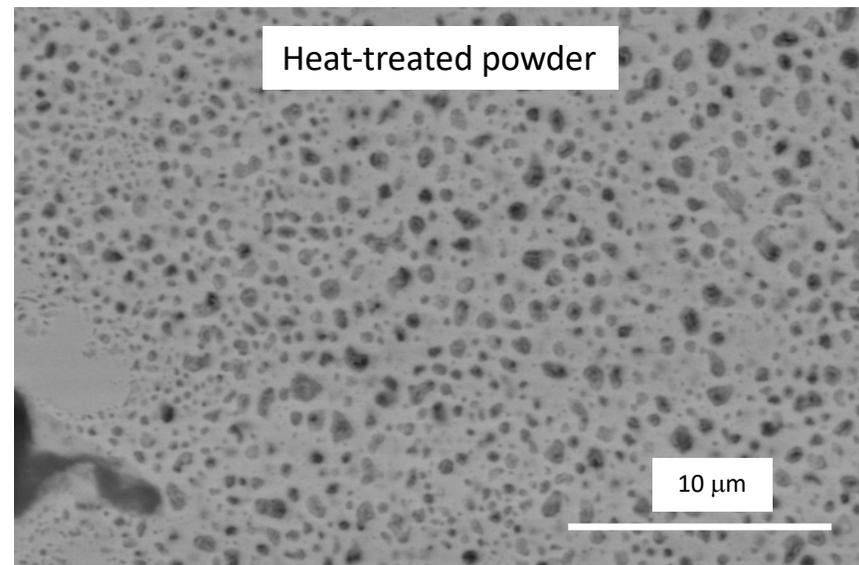
Comparison of Irradiation Behavior of As-Atomized and Heat-Treated U-7Mo Powder: (Irradiated Microstructures)



- Fuel plates EMPI 0702 and EMPI 0821 were irradiated under similar conditions and exhibit similar U-7Mo microstructures

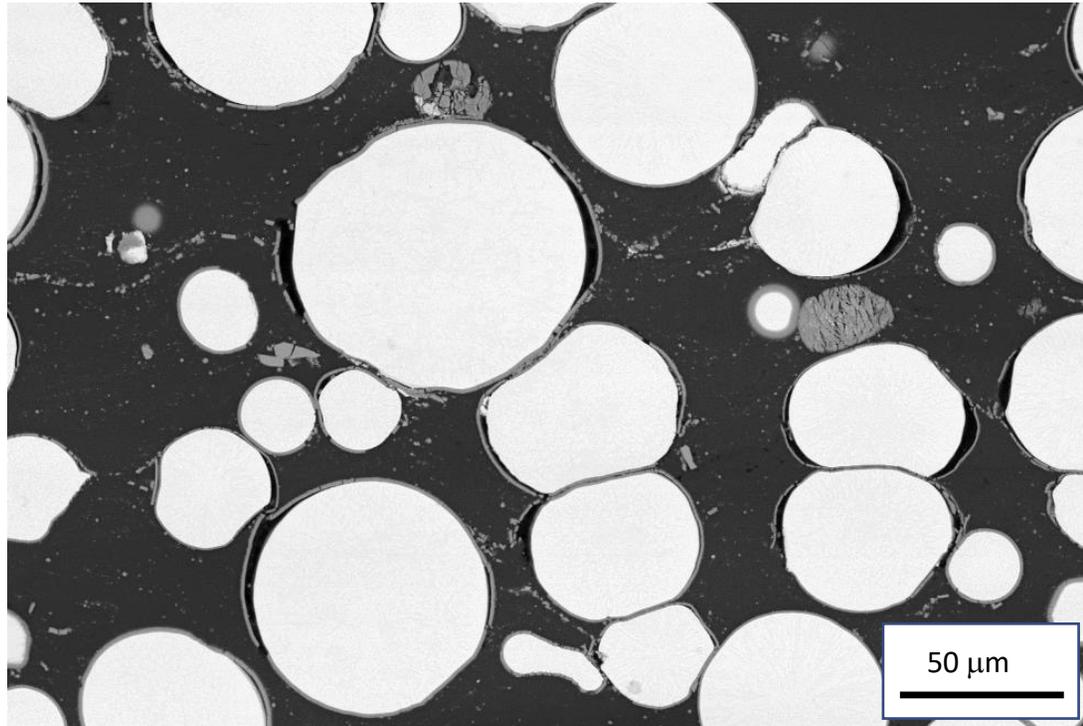


EMPI 0702 (513 W/cm² peak heat flux, 5.7x10²¹ fissions/cm³ average fission density)

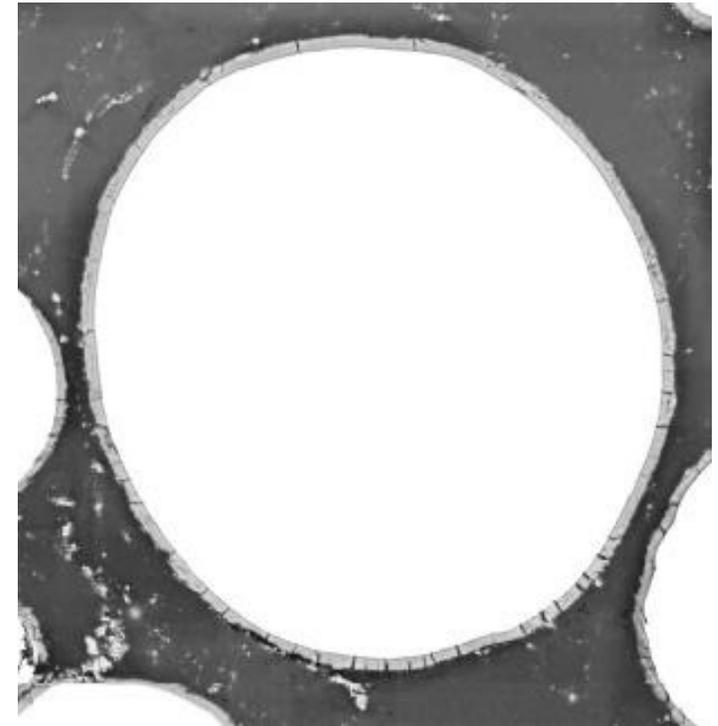


EMPI 0821 (520 W/cm² peak heat flux, 6.3x10²¹ fissions/cm³ average fission density)

Comparison of Irradiation Behavior of Different Batches of ZrN Coating: (As-Fabricated Microstructures)

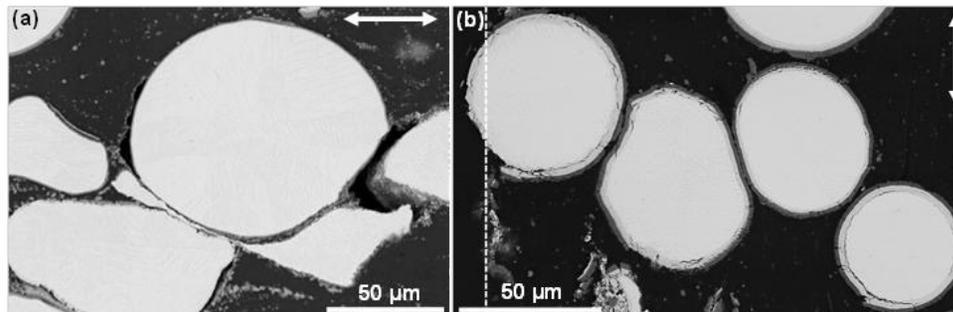


EMPI 0717



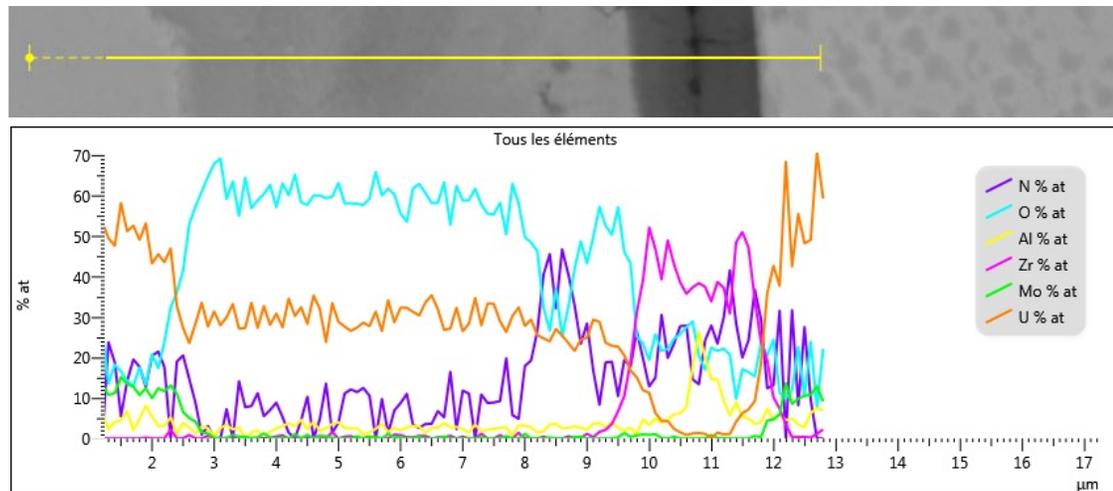
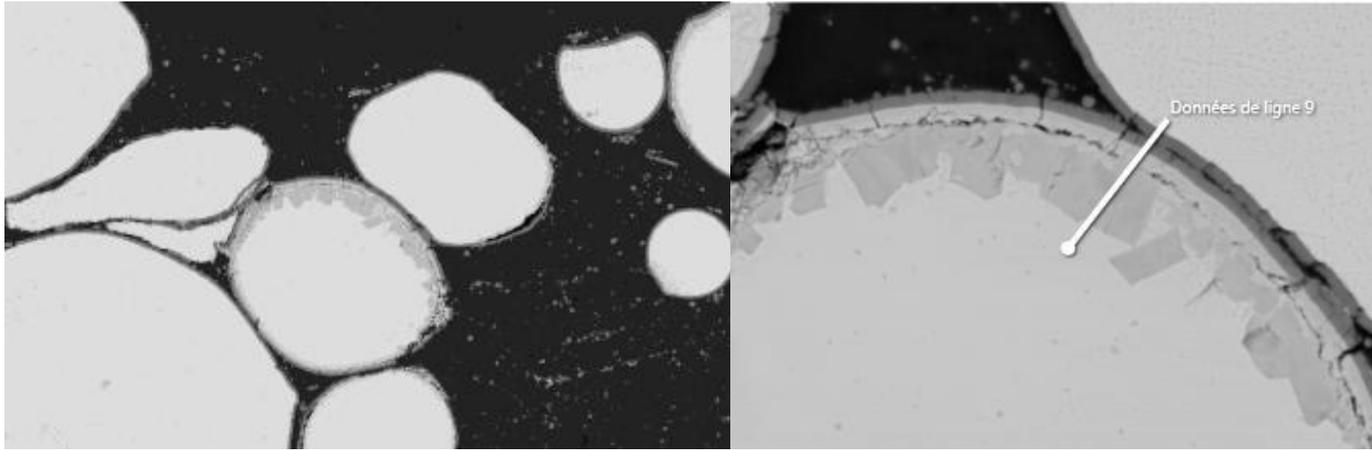
EMPI 0819

EMPI 0828



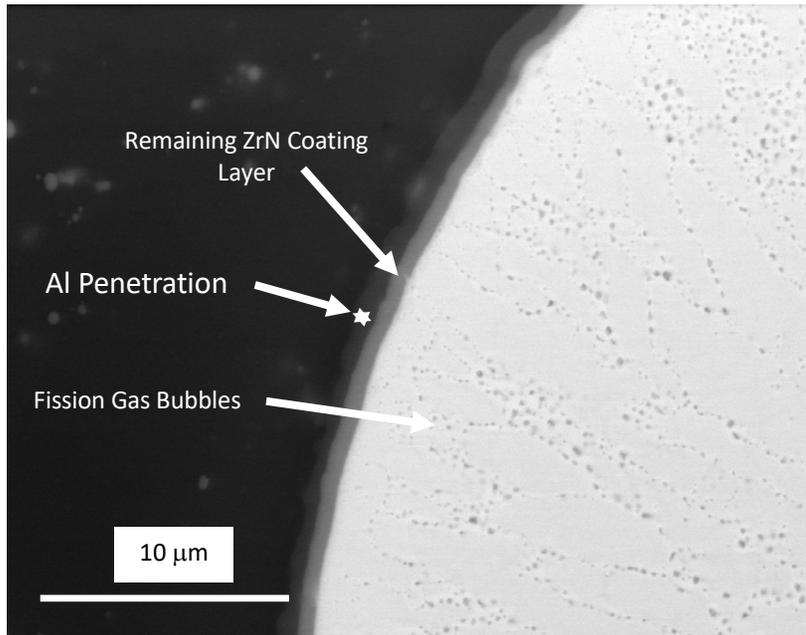
EMPI 0819

Comparison of Irradiation Behavior of Different Batches of ZrN Coating: (As-Fabricated Microstructures)

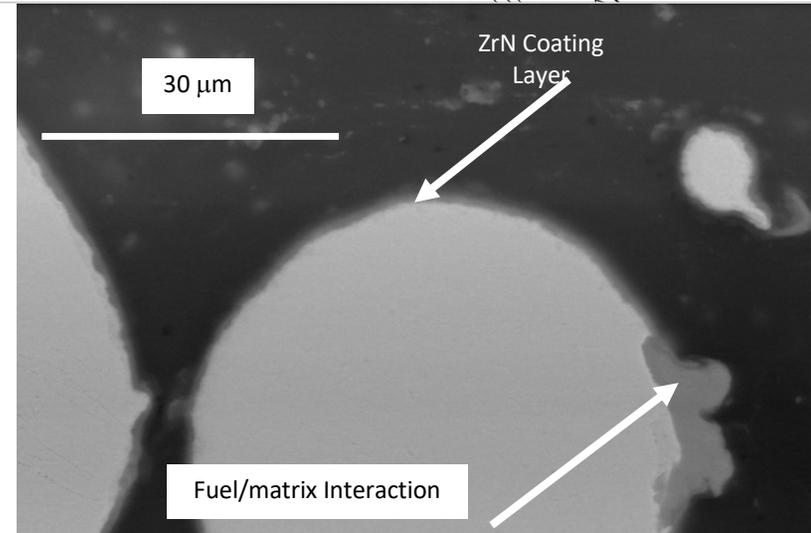


Annealing treatment can oxidize the U-7Mo powders

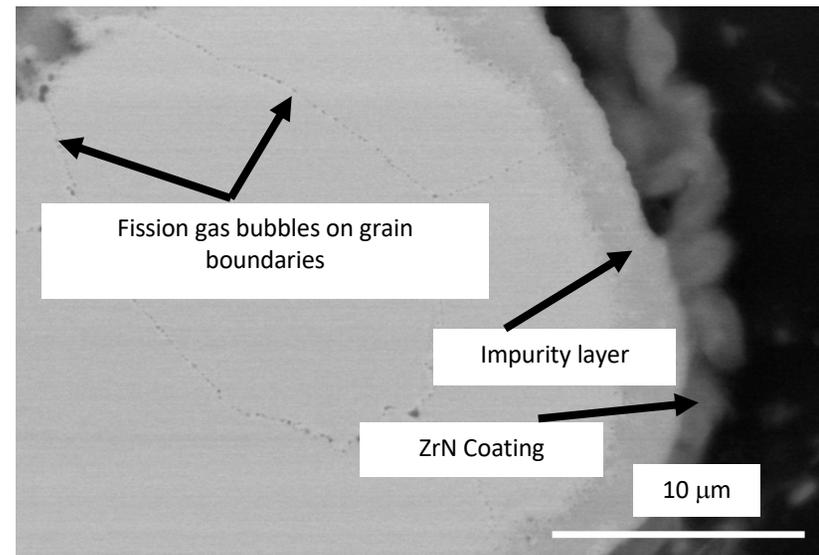
Comparison of Irradiation Behavior of Different Batches of ZrN Coating: (As-Irradiated Microstructures; Medium Fission Density)



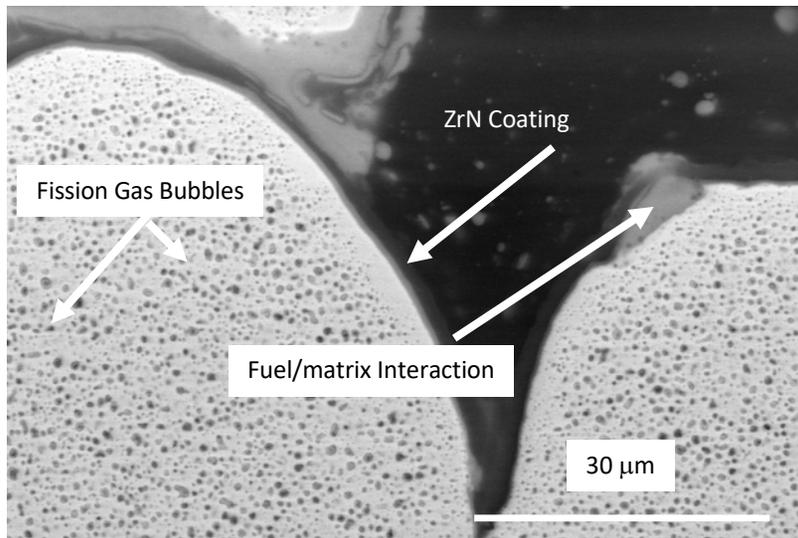
EMPI 0712 (ZrN batch LA3 zero)



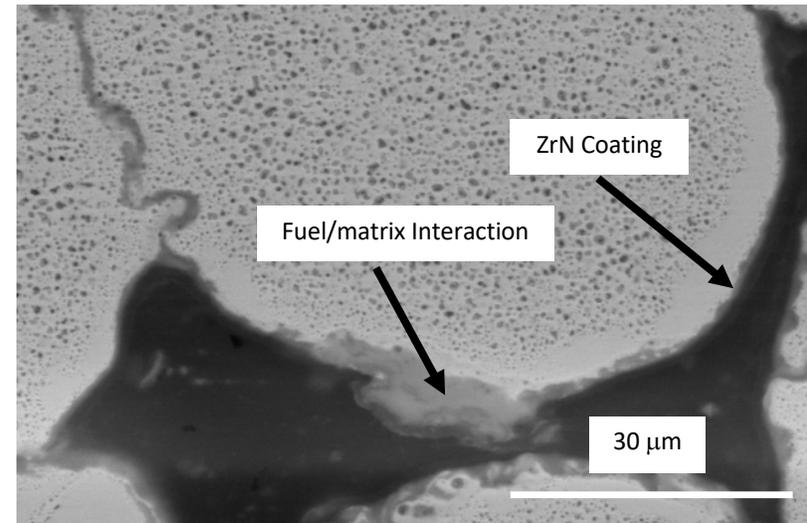
EMPI 0818 (ZrN batch LA4 zero)



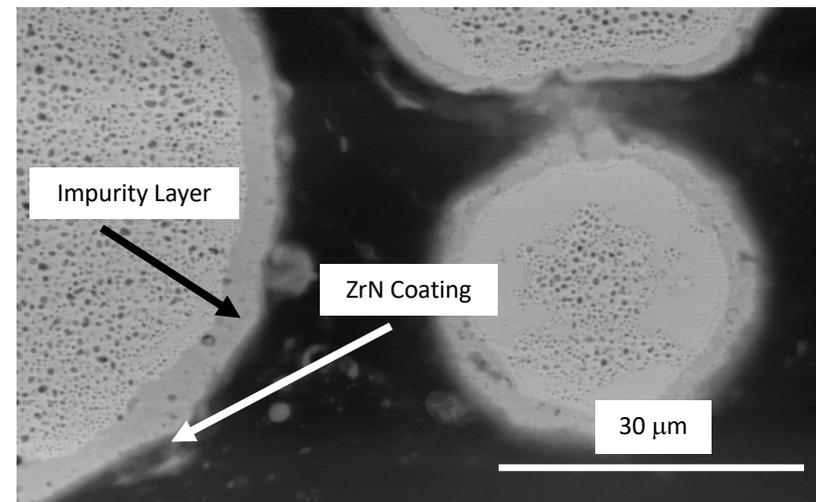
Comparison of Irradiation Behavior of Different Batches of ZrN Coating: (As-Irradiated Microstructures; High Fission Density)



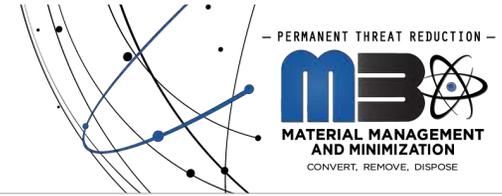
EMPI 0702 (ZrN batch LA3 zero)



EMPI 0821 (ZrN batch LA4 zero)

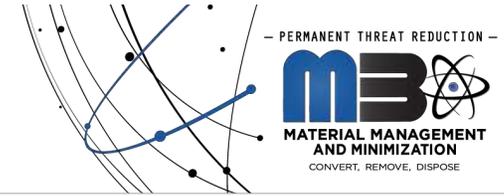


Summary



- SEM imaging completed on the next three highest priority EMPIRE plates.
 - EMPI 0702 and 0712 had PVD ZrN coating and as-atomized U-7Mo powder with no heat treatment, and 0905 used CERCA-produced U-7Mo powder that was heat treated and had PVD ZrN coating.
- EMPI 0905 contained unique phases that may be related to the presence of impurities in the starting fuel meat powders.
- Comparison of Type 7A and 8A fuel plate microstructures is ongoing
 - Similar behavior observed for as-atomized and heat-treated U-7Mo powders irradiated under similar conditions
 - Similar irradiation behavior observed for two different batches of ZrN coatings
 - Image analysis to be performed to quantify any differences

Acknowledgments



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